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पैराफिन वैक्स — विशिष्टि  
( तीसरा पुनरीक्षण )

Paraffin Wax — Specification  
( Third Revision )

ICS 75.140

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## FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Petroleum and Their Related Products of Synthesis or Biological Origin Sectional Committee was approved by the Petroleum, Coal and Related Products Division Council.

Paraffin wax is an important petroleum product manufactured in the country and marketed both at home and abroad. It is produced in several melting point grades and to different quality standards for a variety of applications. This standard is intended to assist in selecting and manufacturing suitable quality of the material for the right purpose.

This standard was first published in 1968 and subsequently revised in 1974 and 1993. Since, then the consumption of paraffin waxes in the country has significantly increased for various end uses apart from traditional use in candle and match manufacturing. In the first revision requirements, such as melting point, oil content and colour were modified, for various end-use trends in the country. In addition ultraviolet absorption test was also been specified for Type I material in addition to acid discolouration test.

The second revision was taken up based on the availability of the paraffin waxes for various end uses. An additional type of paraffin wax Type 2A having oil content 1.5 percent by mass, and widely prevalent in the country has been included. All the existing grades of paraffin wax given in the first revision have been retained. However, the references to their end-uses have been deleted, in order to avoid confusion and leaving it to the individual user to select any suitable grade/type for his specific purpose among the various grades specified. Three amendments were issued to this standard to use Sayolt chromometer as alternate for the loviond colorimeter since, the 457.2 mm (18") cell is no longer manufactured.

In this third revision, the test methods for melting point and determination of oil content are updated. Further, automatic melting point apparatus and temperature measuring devices alternate to mercury thermometer are included. All the three amendments issued to the earlier version are included.

The composition of the Committee responsible for the formulation of this standard is given in Annex G.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rule for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# *Indian Standard*

## PARAFFIN WAX — SPECIFICATION

### ( Third Revision )

#### 1 SCOPE

**1.1** This standard prescribes the requirements and method of sampling and tests for paraffin wax used for waxing paper, making candles, impregnating match sticks and for other general uses.

**1.2** This standard does not cover the requirements of paraffin wax used for explosive and pyrotechnic industry which is covered by a separate standard IS 7401.

#### 2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
266 : 1993	Sulphuric acid
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
1447 (Part 3) : 1992	Methods of sampling of petroleum and its products: Part 3 Methods of sampling of semi-solid and solid petroleum products ( <i>first revision</i> )
1448	Methods of test for petroleum and its products:
[P : 2] : 2007/ ISO 6619 : 1988	Petroleum products and lubricants — Neutralization number — potentiometric titration method ( <i>second revision</i> )
[P : 4/ Sec 1] : 2008/ ISO 6245 : 2001	Petroleum products, Section 1 Determination of ash ( <i>third revision</i> )
[P : 12] : 2013/ ISO 2049 : 1996	Determination of colour (ASTM scale) ( <i>third revision</i> )
[P : 13] : 1960	Colour by lovibond tintometer
[P : 14] : 2019	Methods of test for petroleum and its products: Part 14 Colour by Saybolt chronometer ( <i>first revision</i> )

#### *IS No.*

#### *Title*

[P : 55/Sec 1] : 2004	Determination of saponification value of petroleum products ( <i>first revision</i> )
2480 (Part 1) : 1983	General purpose glass thermometer: Part 1 Solid stem thermometers
7401 : 1987	Paraffin wax for explosives and pyrotechnic industry ( <i>first revision</i> )

#### 3 TYPES AND GRADES

##### 3.1 Types

The material shall be of the following four types:

- a) Type 1 — Extra refined, food grade;
- b) Type 2 — Refined, general purpose paraffin wax;
- c) Type 2A — Refined general purpose paraffin wax; and
- d) Type 3 — Semi-refined.

##### 3.2 Grades

Subject to agreement between the purchaser and the supplier, each of the above three types excluding Type 2A will be graded by its melting point of 2.5 degree spread in the range of 45 to 75°C.

#### 4 REQUIREMENTS

##### 4.1 Description

The material shall essentially be solid saturated hydrocarbons from petroleum, homogeneous in appearance and free from any foreign matter and objectionable odour. It may contain only minimum specified liquid hydrocarbon and shall give on melting, a clear liquid free from water, dirt and other visible impurities.

**4.2** The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in column 7 and 8 of Table 1.

NOTE — It is recognized that there are some applications where, for technical or other reasons, limits different from those specified in this standard or additional requirements may be necessary. The standard does not cater for such special cases, which are for arrangement between the purchaser and the supplier.

## 5 PACKING AND MARKING

### 5.1 Packing

The paraffin wax of Type 1 shall be packed in polyethylene lined new gunny bags. For paraffin waxes Type 2, 2A and 3, new gunny bags / HDPE woven sacks or barrels as agreed to between the purchaser and the supplier shall be used.

### 5.2 Marking

**5.2.1** Each container shall marked with the following information:

- Name, type, grade and mass of the material;
- Indication of the source of manufacture, recognized trademark, if any;
- Batch or code number or otherwise to enable the lot of consignment/or manufacture to be traced back for records;
- Containers for Type 2, 2A and 3 paraffin wax shall be labelled conspicuously with the words:  
“NOT TO BE USED FOR FOOD PACKAGING,

COSMETICS PREPARATION OR MEDICINAL USE”; and

- Any other statutory requirements.

**5.2.2** The material shall also be marked with its melting point.

### 5.2.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark

## 6 SAMPLING

**6.1** Representative samples of the material shall be drawn as prescribed in IS 1447 (Part 3).

**6.2** Test on melting point (cooling curve) shall be conducted on individual samples and the rest of the tests shall be conducted on composite samples.

**Table 1 Requirements for Paraffin Wax**  
( Clause 4.2 )

Sl No.	Characteristics	Requirement for				Method of Test, Ref to	
		Type 1	Type 2	Type 2A	Type 3	Annex	IS 1448
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Melting point (cooling curve)	← 45 to 75°C →		58°C Min.	45 to 75°C	A	—
ii)	Ash, percent by mass, <i>Max.</i>	0.03	0.03	0.03	0.03	—	[P : 4/ Sec 1]
iii)	Acidity						[P : 2]
	a) Organic, (mg of KOH/gm), <i>Max.</i>	0.10	0.10	0.10	0.10	—	
	b) Inorganic, (mg of KOH/gm), <i>Max.</i>	0.02	0.02	0.02	0.02	—	
iv)	Saponification value, <i>Max</i>	1.0	1.0	1.0	1.0	—	[P : 55]
v)	Oil content, percent by mass, <i>Max</i>	0.50	0.50	1.50	3.50	B	—
vi)	Colour						
	a) Lovibond in 457.2 mm (18 inch) cell, <i>Max</i>	1.0Y	2.0Y	2.0Y	—	—	[P : 13] Method A
	b) Saybolt, <i>Min</i>	+24	+20	+15	—	—	[P : 14]/ASTM D6045
	c) ASTM, <i>Max</i>	—	—	—	2.0	—	[P : 12]
vii)	Acid discolouration test	To pass the test	—	—	—	C	—
viii)	Ultra violet (UV) Absorbance per centimetre path length, <i>Max</i>					D	—
	280 - 289 nm	0.15	—	—	—		
	290 - 299 nm	0.12	—	—	—		
	300 - 359 nm	0.08	—	—	—		
	360 - 400 nm	0.02	—	—	—		
ix)	Odour, <i>Max</i>	1	—	—	—	E	—

#### NOTES:

**1** When ASTM colorimeter is not available, the colour may be determined by Lovibond Tintometer in accordance with method A in [P : 13] of IS 1448, using 6.34 mm (¼ inch) cell. The corresponding colour requirements are -4.5Y +0.4R, *Max.*

**2** For Type 1, 2 and 2A, colour may be tested either by (a) or (c). In case of any dispute, referee method is [P : 14].

## ANNEX A

[ Table, 1, Sl No. (i) ]

## DETERMINATION OF MELTING POINT (COOLING CURVE)

## A-1 GENERAL

**A-1.1** Melting point (cooling curve) is a test that is, widely used by wax suppliers and consumers. It is particularly applied to petroleum waxes that are rather highly paraffinic or crystalline in nature. A plateau occurs with specimens containing appreciable amounts of hydrocarbons that crystallize at the same temperature, giving up heat of fusion, thus temporarily retarding the cooling rate. In general, petroleum waxes with large amounts of non-normal hydrocarbons or with amorphous solid forms do not show the plateau.

**A-1.2** This method describes a procedure for determination of the melting point (cooling curve) of petroleum wax. It is unsuitable for waxes of the petrolatum group, the petroleum ceresin group, or to blend of such waxes with paraffin wax or scale.

## A-2 TERMINOLOGY

For the purpose of this test, the following definition shall apply.

**A-2.1 Melting Point (Cooling Curve) of Petroleum Wax**

The temperature at which petroleum wax first shows a minimum rate of temperature change when allowed to cool under prescribed conditions.

## A-3 OUTLINE OF THE METHOD

**A-3.1** In procedure A (manual method), a specimen of molten wax in a test-tube fitted with a thermometer or equivalent temperature measuring device is placed in an air-bath, which in turn is surrounded by a water bath held at 16 to 28°C. As the molten wax cools, periodic readings of its temperature are taken. When solidification of the wax occurs and a minimum rate of temperature change (cooling curve) is observed, the temperature at that point is recorded as the melting point (cooling curve) of the sample.

**A-3.2** In procedure B, an automatic analyzer is used. As the molten wax cools, the sample temperature decrease is measured every 15s in 0.01°C readings. The melting point is considered to be reached when five consecutive measurements are constant within a given temperature interval, usually 0.1°C.

## A-4 APPARATUS

**A-4.1 Test Tube**

Outside diameter of test tube is 25 mm and 100 mm

in length. It may be marked with a filling line 51 mm above the bottom.

**A-4.2 Air Bath**

Inside diameter of 51 mm and 114 mm in depth having, a cork, with a hole for holding, the test tube firmly in a vertical position in its centre.

**A-4.3 Water Bath**

A suitable cylindrical vessel 130 mm in inside diameter and 152 mm in depth. A fitted cover to support the air bath vertically shall be provided so that the sides and bottom of the air bath are surrounded by a layer of water 38 mm thick. The cover shall have an opening at a distance of 19 mm from the outer wall of the water bath for suspending the bath thermometer. The air bath and water bath cover may be made in one assembly as shown in Fig.1.

**A-4.4 Thermometers**

**A-4.4.1** Water bath thermometer shall be of any suitable type [see IS 2480 (Part 1)], accurate to 1°C throughout the required range.

**A-4.4.2** For noting the melting point of wax, another thermometer complying with the following requirements shall be used:

Range	38 to 82°C
Graduation	0.1°C
Immersion	79 mm
Overall length	365 ± 10 mm
Stem diameter	5.5 to 8.0 mm
Bulb shape	Cylindrical
Bulb length	18 to 20 mm
Bulb diameter	5.0 to 8.0 mm
Length of graduated portion	215 to 240 mm
Distance between bottom of bulb to minimum graduation	105 to 115 mm
Longer lines at each	0.5°C
Figured at each	1°C
Expansion chamber	To allow heating to 100°C
Top finish	Plain
Scale error not to exceed	± 0.1°C

NOTE — IP 17C thermometers also conform to these requirements.

**A-4.5 Temperature Measuring Devices**

Other than those described in A-4.4, such as resistance thermometer are satisfactory for this test method, if they exhibit the same temperature response as the equivalent mercury-in-glass thermometers.

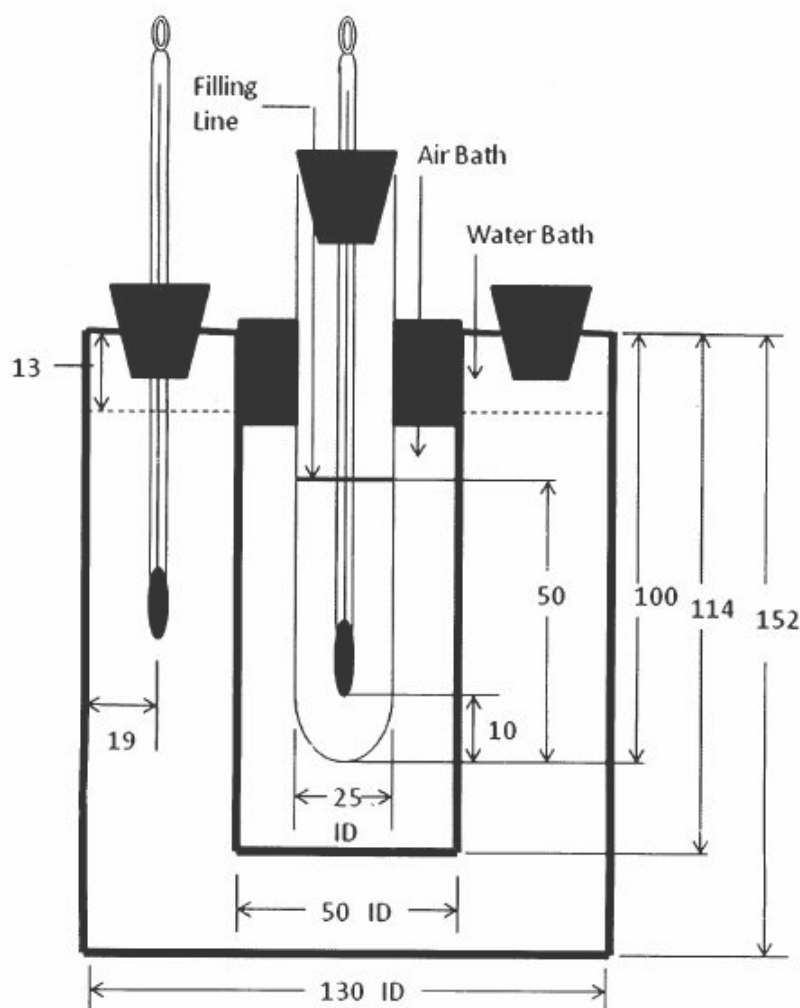
**A-4.6** The automatic instrument consists of a bath (for example, an aluminum block with two measuring locations, two apertures to place the test tubes, and two apertures for the temperature probes). The apparatus may have an accessory digital display and a printer. The temperature of the bath is maintained between 22°C and 26°C. A heating device is used to increase the temperature, and a cooling device with cold water circulation is used to decrease the temperature. The temperature may be monitored with a calibrated thermometer or an electronic temperature-measuring device of equivalent precision and accuracy.

**A-5 PROCEDURE A (MANUAL METHOD)**

**A-5.1** Assemble the apparatus as shown in Fig. 1. Fill the water bath to within 13 mm of the top with water at a temperature of 16 to 28°C. The bath temperature shall be maintained within these limits throughout the test.

**A-5.2** Heat the wax sample to at least 8°C above its melting point by taking it in a test tube, kept in a water-bath, maintained at a temperature of not more than 20°C above the expected melting point of the wax.

**A-5.3** Fill the test tube to a height of 51 mm with the molten sample. Insert the melting point thermometer through the centre of a cork in such a manner that the distance between the bulb of the thermometer and the bottom of test tube is 10 mm. Keep the test tube assembly in the air bath while the temperature of molten wax in the test tube is still at least 8°C above its melting point.



All dimensions in millimeters

FIG. 1 ASSEMBLY OF APPARATUS FOR DETERMINATING MELTING POINT (COOLING CURVE) OF PETROLEUM WAX

**A-5.4** Read the melting thermometer reading after every 15 s. Discontinue the test when five consecutive readings which agree within  $0.1^{\circ}\text{C}$ , have been obtained or at least 3 min after the temperature begins to fall following a series of readings agreeing with  $0.1^{\circ}\text{C}$ .

#### **A-6 PROCEDURE B (AUTOMATIC METHOD)**

**A-6.1** Place a clean test tube held in a polytetrafluoroethylene (PTFE) holder ring in the aperture provided in the apparatus.

**A-6.2** Insert the temperature probe into a centrally bored, one-holed stopper, and insert it in the test tube. Check the probe height to reach manufacturer's suggested height. Place the stopper with the probe back in the resting holder provided.

**A-6.3** Bring the sample to a temperature at least  $8^{\circ}\text{C}$  above the expected melting point. Heat the sample in a water bath, with  $93^{\circ}\text{C}$  maximum temperature.

**A-6.4** Add the molten sample to the test tube to the filling mark. Place the stopper with the probe on the test tube assembly. Insert the assembly into the aluminum block aperture, and initiate the analysis in accordance with the manufacturer's instructions.

**A-6.5** When the melting point is detected, the analysis will automatically stop. The resulting melting point will be displayed on the digital monitor, or printed on a printer, or both, as per the options available in the instrument.

#### **A-7 CALCULATION AND REPORTING**

**A-7.1** Average the five consecutive thermometer readings, which include the least temperature, change. Correct this average for error in thermometer scale, if necessary.

**A-7.2** Report the result to the nearest  $0.1^{\circ}\text{C}$  as the petroleum wax melting point (cooling curve).

#### **A-8 PRECISION**

Results of duplicate tests shall not differ by more than the following amounts:

- |                           |  |                        |
|---------------------------|--|------------------------|
| a) <i>Repeatability</i>   |  |                        |
| 1) Manual apparatus       |  | $0.11^{\circ}\text{C}$ |
| 2) Automatic apparatus    |  | $0.23^{\circ}\text{C}$ |
| b) <i>Reproducibility</i> |  |                        |
| 1) Manual apparatus       |  | $0.41^{\circ}\text{C}$ |
| 2) Automatic apparatus    |  | $0.50^{\circ}\text{C}$ |

## ANNEX B

[ Table 1, Sl No. (v) ]

## DETERMINATION OF OIL CONTENT

## B-1 GENERAL

This method describes a test procedure for the determination of oil content in petroleum waxes having a congealing point of 30°C or higher and containing not more than 15 percent of oil.

## B-2 OUTLINE OF THE METHOD

The sample is dissolved in methyl ethyl ketone. The solution cooled to -32°C to precipitate the wax, and filtered. The oil content of the filtrate is determined by evaporating the methyl ethyl ketone and weighing the residue.

## B-3 APPARATUS

## B-3.1 Filter Sticks and Assembly

Consisting of a 10 mm diameter sintered glass filter stick of 10 to 15  $\mu$  maximum pore diameter as determined by the method given in Annex F. It consists of an air

pressure inlet tube, delivery nozzle and a ground glass joint to fit a 25  $\times$  170 mm test tube. The dimensions for a suitable filtration assembly are shown in Fig. 2.

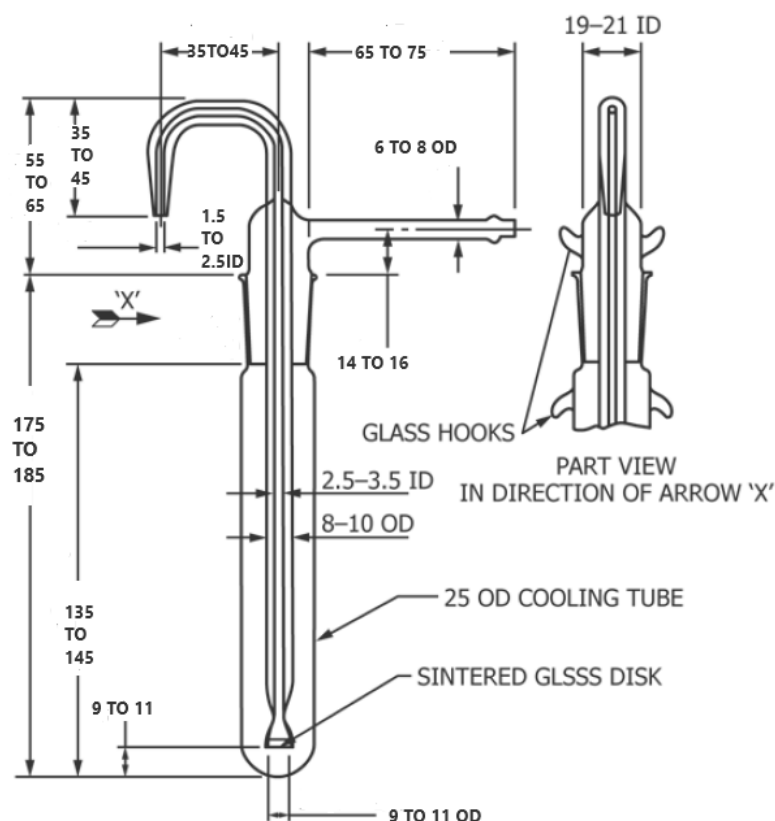
NOTE — A metallic filter stick may be employed, if desired. A filter stick made of stainless steel and having a 12.7 mm disc of 10 to 15  $\mu$  maximum pore diameter has been found to be satisfactory. The metallic apparatus is inserted into a 25  $\times$  150 mm test tube and held in place by means of a cork.

## B-3.2 Cooling Bath

Consisting of an insulated box with 30 mm holes in the centre to accommodate any desired number of test tubes. The bath may be filled with a suitable medium, such as kerosene, and may be cooled by circulating a refrigerant through coils, or by using solid carbon dioxide. A suitable cooling bath to accommodate three test tubes is shown in Fig. 3.

## B-3.3 Dropper Pipette

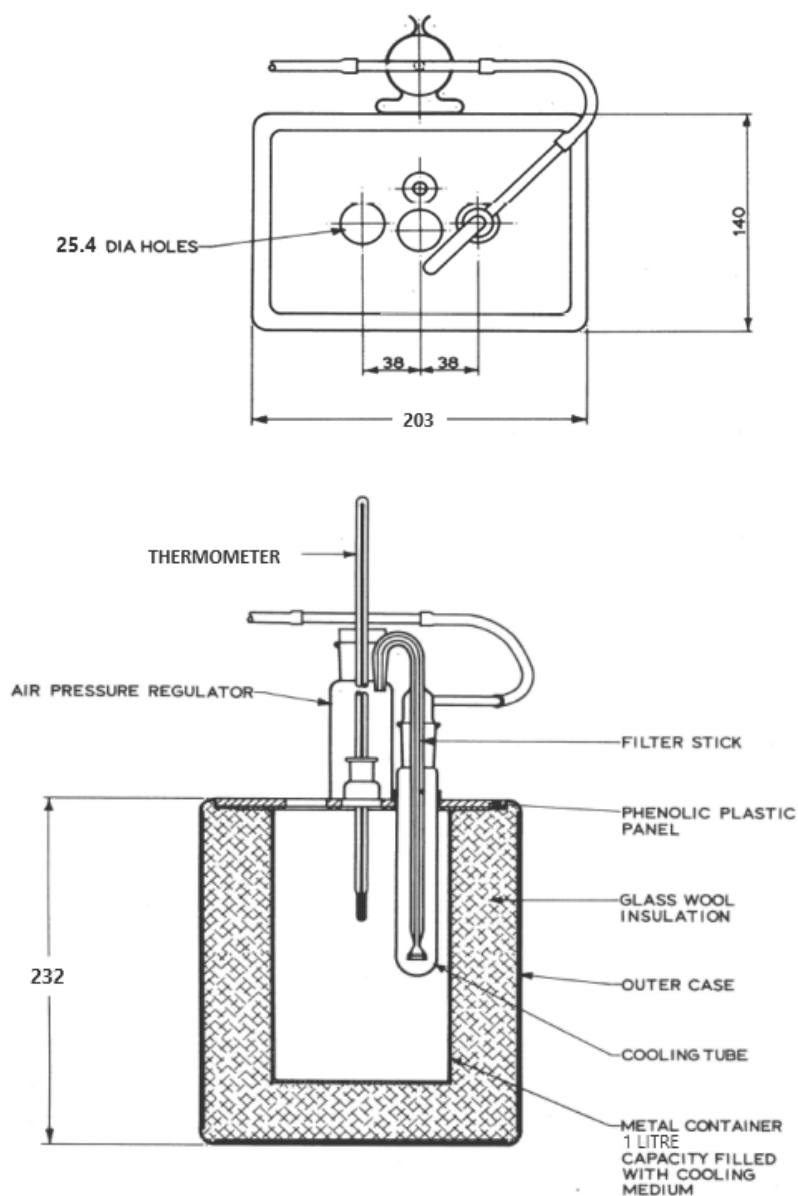
Provided with a rubber bulb and calibrated to deliver  $1 \pm 0.05$  g of molten wax.



All dimensions in millimeters

FIG. 2 FILTER STICK





All dimensions in millimeters.

FIG. 3 COOLING BATH

### B-3.4 Transfer Pipette

Calibrated to deliver  $15 \pm 0.06$  ml.

### B-3.5 Air Pressure Regulator

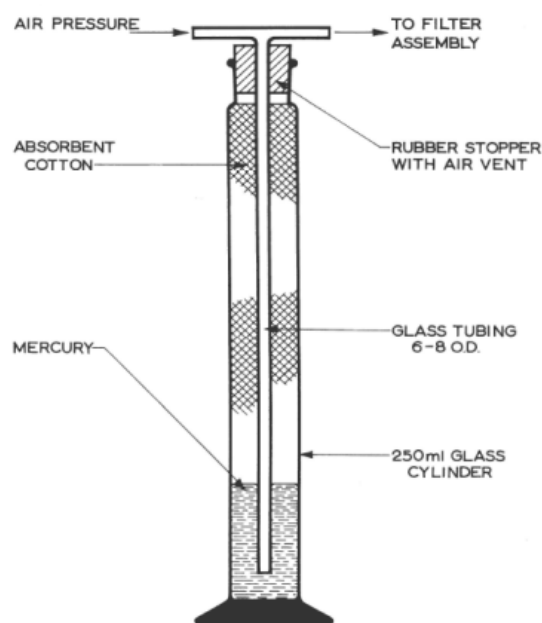
Designed to supply air to the filtration assembly (see B-6.5) at the volume and pressure required to give an even flow of filtrate. Either the conventional pressure reducing valve or a mercury bubbler type regulator are found satisfactory. The latter type, illustrated in Fig. 4 consists of a 250 ml glass cylinder and a T-tube held in the cylinder by means of a rubber stopper grooved at the sides to permit the escape of excess air. The volume and pressure of the air supplied to the filtration assembly is regulated to a depth to

which the T-tube is immersed in mercury at the bottom of the cylinder. Absorbent cotton placed in the space above the mercury prevents the loss of mercury by spattering. The air pressure regulator is connected to the filter stick and assembly by means of rubber tubing.

### B-3.6 Thermometer

Conforming to schedule mark 3 of IS 2480 (Part 1). Temperature measuring devices other than this are satisfactory, if they exhibit the same temperature response as the equivalent mercury-in-glass thermometers.

NOTE — IP 72C thermometers also conform to the requirement.



All dimensions in millimeters

FIG. 4 AIR PRESSURE REGULATOR

### B-3.7 Weighing Bottles

Conical in shape and glass stoppered, having a capacity of 15 to 25 ml.

### B-3.8 Evaporation Assembly

Consisting of an evaporating cabinet and connections, essentially as illustrated in Fig. 5, and capable of maintaining a temperature of  $35 \pm 1^\circ\text{C}$  around the evaporation flasks. Construct the jets with an inside diameter of  $4 \pm 0.2$  mm for delivering a stream of clean, dry air vertically downward into the weighing bottle. Support each jet so that the tip is  $15 \pm 5$  mm above the surface of the liquid at the start of the evaporation. Supply air at the rate of 2 to 3 litres per min per jet purified by passage through a tube of 1 cm bore packed loosely to a height of 20 cm with absorbent cotton. Periodically check the cleanliness of the air by evaporating 4 ml of methyl ethyl ketone by the procedure specified in B-6.6. When the residue does not exceed 0.1 mg, the evaporation equipment is operating satisfactorily.

### B-3.9 Analytical Balance

Capable of reproducing weights to 0.1mg. The sensitivity should be adjusted so that 0.1 mg will deflect the pointer one-half division on the pointer scale.

### B-3.10 Wire Stirrer

A piece of stiff iron or nichrome wire of about 1 mm diameter, 250 mm long. A 10 mm diameter loop is formed at each end, and the loop at the bottom end is

bent so that the plane of the loop is perpendicular to the wire.

## B-4 SOLVENT

### B-4.1 Methyl Ethyl Ketone

Distilling between  $78$  to  $81^\circ\text{C}$  and conforming to following requirements. The material shall be stored over anhydrous calcium sulphate (5 percent by mass of the solvent) and shall be filtered before use.

Characteristic	Requirement
Colour	Water white
Relative density, $20/20^\circ\text{C}$	$0.805 - 0.807$
Acidity, as acetic acid, percent by mass, <i>Max.</i>	0.003
Water content, percent by mass	0.3
Refractive index at $20^\circ\text{C}$	$1.378 \pm 0.002$
Residue on evaporation of 4 ml of sample, mg, <i>Max.</i>	0.1

NOTE — Residue on evaporation may be found out by the procedure described in B-6.6.

## B-5 SAMPLE

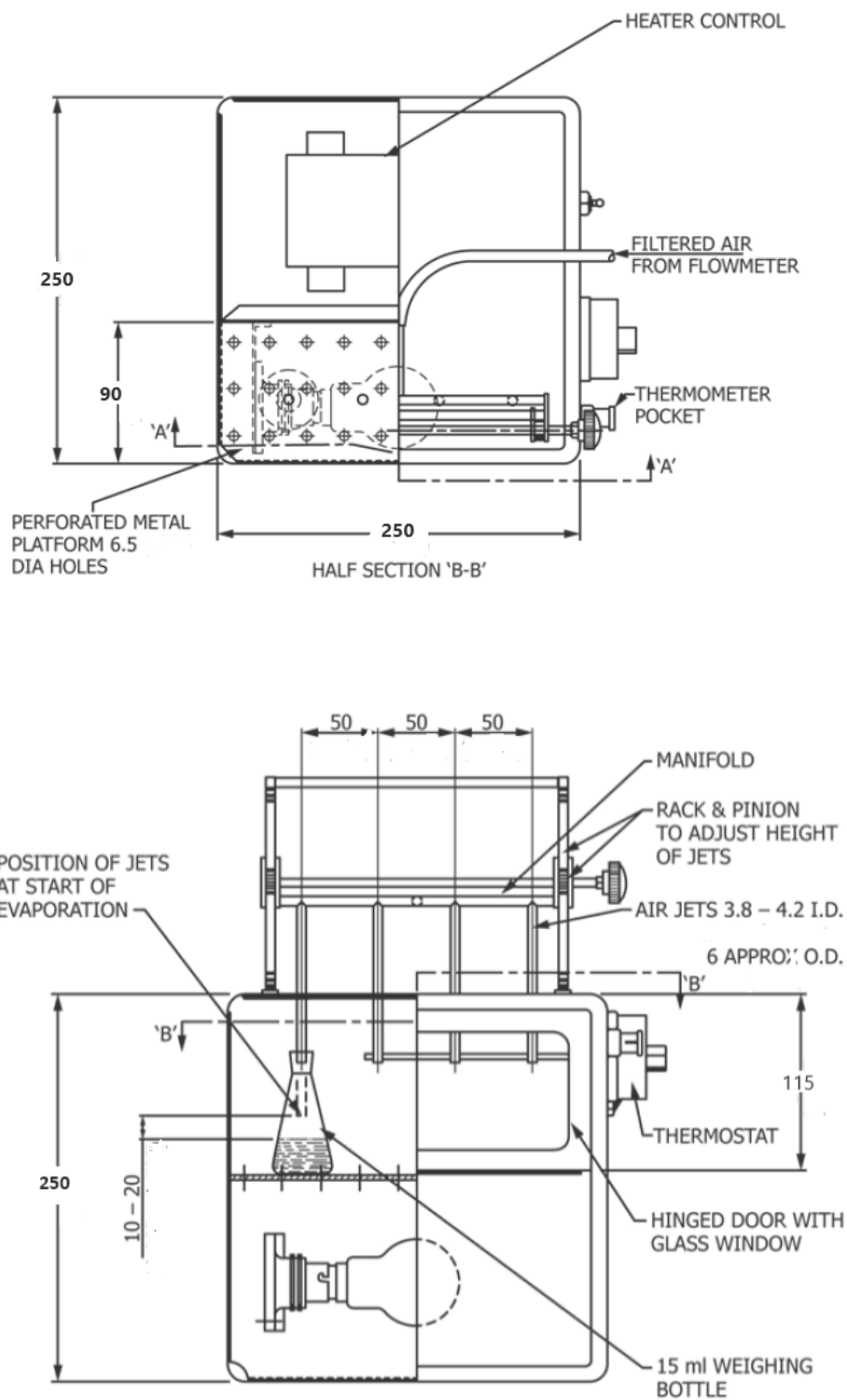
If the sample of wax is 1 kg or less, obtain a representative portion by melting the entire sample and stirring thoroughly. For samples over 1 kg, exercise special care to ensure obtaining a truly representative portion, bearing in mind that the oil may not be distributed uniformly throughout the sample, and that mechanical operations may express some of the oil.

## B-6 PROCEDURE

**B-6.1** Melt a representative portion of the sample in a beaker, using a water bath or oven maintained at  $70$  to  $100^\circ\text{C}$ . As soon as the wax is completely melted, thoroughly mix by stirring. Preheat the dropper pipette in order to prevent the solidification of wax in the tip, and withdraw a 1 g portion of the sample as soon as possible after the wax has melted. Hold the pipette in a vertical position, and carefully transfer its contents into a clean, dry test tube, previously weighed to the nearest 1 mg (*see* Note). Swirl the test tube so as to coat the bottom evenly with wax. This permits more rapid solution latter. Allow the test tube to cool, and weigh to the nearest 1 mg.

NOTE — The mass of a test tube, which is cleaned by means of solvents, does not vary to a significant extent. Therefore, a tare mass may be obtained and used repeatedly.

**B-6.2** Pipette 15 ml of methyl ethyl ketone into the test tube and place the latter just up to the level of its contents in a hot water or steam bath. Heat the solvent-



### HALF SECTION AA

All dimensions in millimeters

FIG. 5 EVAPORATION ASSEMBLY

wax mixture, stirring up and down with the wire stirrer, until a homogeneous solution is obtained. Exercise care to avoid loss of solvent by prolonged heating

NOTE — Very high-melting wax samples may not form clear solution. Stir until the undissolved material is well dispersed as a fine cloud -.

**B-6.3** Plunge the test tube into a 800 ml beaker of ice water and continue to stir until contents are cold. Remove the stirrer. Remove the test tube from the ice bath, wipe dry on the outside with a cloth and weigh to the nearest 0.1 g. During this operation, solvent shall not be allowed to vaporize by more than 1 percent. The mass of solvent is, therefore, kept practically a constant and after a few samples are weighed, this mass, approximately 11.9 g, may be used as a constant factor.

**B-6.4** Place the test tube containing the wax solvent slurry in the cooling bath, which is, maintained at  $-34.5 \pm 1.0^\circ\text{C}$ . During this chilling operation, it is important that stirring by means of the thermometer be almost continuous, in order to maintain a slurry of uniform consistency as the wax precipitates. Do not allow the wax to set up on the walls of the cooling vessel nor permit any lumps of wax crystals to form. Continue stirring until the temperature reaches  $-31.7 \pm 0.3^\circ\text{C}$ .

**B-6.5** Immerse in the mixture, the clean, dry filter stick, which has previously been cooled by placing it in a test tube and holding at  $-34.5 \pm 1^\circ\text{C}$  in the cooling bath for a minimum of 10 min. Seat the ground glass joint of the filter to make an airtight seal. Place an unstoppered weighing bottle, previously weighed together with glass stopper to the nearest 0.1 mg, under the delivery nozzle of the filtration assembly.

NOTE — Take every precaution to ensure the accuracy of the mass of the stoppered weighing bottle prior to determining this mass, rinse and clean the weighing bottle with methyl ethyl ketone, dry, and stopper, wipe dry on the outside with a cloth, and place in the evaporation assembly to dry for about 5 min. Then remove the weighing bottle and stopper. Place near the balance, and allow standing for 10 min prior to weighing. Stopper the bottle during this cooling period. Once the weighing bottle and stopper have been dried in the evaporation assembly, lift only with forceps. Take care to remove and replace the glass stopper with a light touch.

**B-6.6** Apply air pressure to the filtration assembly, and immediately collect about 4 ml of filtrate in the weighing bottle. Release the air pressure to permit the liquid to drain back slowly from the delivery nozzle.

Remove the weighing bottle immediately, stopper, and weigh to the nearest 10 mg without waiting for it to cool down to room temperature. Unstopper the weighing bottle and place it under one of the jets in the evaporation assembly maintained at  $35 \pm 1^\circ\text{C}$  with the air jet centered inside the neck, and the tip  $15 \pm 5$  mm above the surface of the liquid. After the solvent has evaporated, which usually takes less than 30 min, remove the bottle, stopper, and place near the balance. Allow to stand for 10 min and weigh to the nearest 0.1 mg.

**B-6.6.1** Repeat the evaporation procedure using a 5 min evaporation period instead of 30 min, until the loss between successive weighing it not over 0.2 mg.

## B-7 CALCULATION

Calculate the amount of oil in the wax as follows.

$$\text{Oil content, percent by mass} = \frac{100 AC}{BD} - 0.15$$

Where,

A = mass, in g of oil residue;

B = mass, in g of wax sample;

C = mass, in g of solvent obtained by subtracting mass of test tube plus wax sample (**B-6.1**) from mass of test tube and contents (**B-6.2**);

D = mass, in g of solvent evaporated, obtained by subtracting the mass of weighing bottle plus oil residue from mass of weighing bottle plus filtrate (**B-6.5**); and

0.15 = the average correction factor for the solubility of wax in the solvent at  $-31.7^\circ\text{C}$

## B-8 REPORTING

Report the result as oil content, percent by mass, if the result is negative, report as zero.

## B-9 PRECISION

Results of duplicate tests shall not differ by more than the following amounts:

<i>Repeatability</i>	<i>Reproducibility</i>
0.06 + 8 percent of the mean	0.2 +11 percent of the mean

## ANNEX C

[Table, 1, Sl No. (vii)]

### ACID DISCOLOURATION TEST

#### C-1 SCOPE

This method is indicative of the degree of refining of paraffin wax and enables the quality of wax used for food packaging.

#### C-2 APPARATUS

**C-2.1** In addition to the apparatus prescribed in IS 1448 [P : 13], the apparatus prescribed in **C-2.2** and **C-2.3** is required. In the tintometer, 6.35 mm (¼ inch) cell shall be used for the determination of acid color after acid discoloration test.

#### C-2.2 Test Tube

Made of heat resistant glass, 200 mm and 30 mm in diameter.

#### C-2.3 Stirrer

Made of glass, with a 7 mm diameter glass rod, 200 mm in length fitted with a 7 x 20 mm straight paddle at the end. It shall be driven by an electric motor at a speed of about 1 500 rev/min.

#### C-3 REAGENT

Sulphuric acid, 90 percent (*see* IS 266).

#### C-4 PROCEDURE

**C-4.1** Place 10 g of the sample in a clean and dry test tube. Place the tube in a water bath maintained at 90°C, about half the test tube being immersed. After 10 min, add 10 ml of sulphuric acid from a measuring cylinder to the test tube. Stir the mixture with the glass stirrer at about 1 500 rev/min. After 5 min remove the stirrer and allow the contents of the tube, still in the water bath, to settle for another 5 min. Remove the tube from bath and cool to allow the wax to congeal. Pierce a hole through the wax by a glass rod, pour the acid into the 6.35 mm (¼ inch) tintometer cell, and measure the tint.

**C-4.2** The materials shall be regarded to have passed the test if Lovibond colour in 6.35 mm (¼ inch) cell of acid, after acid discoloration test does not exceed 2.0 (yellow series 510), 1.0 (Red series 200).

## ANNEX D

[ Table 1, Sl No. (viii) ]

### ULTRA VIOLET ABSORBANCE TEST

#### D-1 GENERAL INSTRUCTIONS

Because of the sensitivity of the test, the possibility of error arising from contamination is great. It is of the greatest importance that all glassware be scrupulously cleaned to remove all organic matter, such as oil, grease, detergent residues, etc. Examine all glassware including stoppers and stopcocks, under ultraviolet light to detect any residual fluorescent contamination. As a precautionary measure, it is recommended practice to rinse all glassware with purified iso octane immediately before use. No grease is to be used on stopcocks or joints. Great care to avoid contamination of wax samples in handling and to assure absence of any extraneous material arising from inadequate packaging is essential. Because some of the polynuclear hydrocarbons sought in this test are very susceptible to photo oxidation, the entire procedure is to be carried out under subdued light.

#### D-2 APPARATUS

##### D-2.1 Separatory Funnels

Funnels of 250 ml, 500 ml, 1 000 ml and preferably 2 000 ml capacity, equipped with tetrafluoroethylene polymer stopcocks.

##### D-2.2 Reservoir

Reservoir of 500 ml capacity, equipped with a 24/40 standard taper male fitting at the bottom and a suitable ball-joint at the top for connecting to the nitrogen supply. The male fitting should be equipped with glass hooks.

##### D-2.3 Chromatographic Tube

Chromatographic tube of 180 mm in length, 15.7 ± 0.1 mm inside diameter, equipped with a coarse, fritted glass disc, a tetrafluoroethylene polymer stopcock, and

a female 24/40 standard tapered fitting at the opposite end (overall length of the column with the female joint is 235 mm). The female fitting should be equipped with glass hooks.

#### D-2.4 Disc

Tetrafluoroethylene polymer 50.8 mm diameter disc approximately 4.76 mm thick with a hole bored in the center to closely fit the stem of the chromatographic tube.

#### D-2.5 Heating Jacket

Conical for 500 ml separating funnel (used with variable transformer heat control).

#### D-2.6 Suction Flask

250 ml or 500 ml filter flask.

#### D-2.7 Condenser

24/40 joints, fitted with a drying tube, length optional

#### D-2.8 Evaporation Flask (Optional)

250 ml or 500 ml capacity all glass flask equipped with standard taper stopper having inlet and outlet tubes to permit passage of nitrogen across the surface of contained liquid to be evaporated.

#### D-2.9 Vacuum Distillation Assembly

All glass (for purification of dimethyl sulphoxide) 2 litre distillation flask with heating mantle; vacuum-jacketed condenser (or equivalent) about 45 cm in length and distilling head with separable cold finger condenser. Use of tetrafluoroethylene polymer sleeves on the glass joints will prevent freezing. Do not use grease on stopcocks or joints.

#### D-2.10 Spectrophotometric Cells

Fused quartz cells, optical path length in the range of  $5.000 \pm 0.005$  cm; also for checking spectrophotometer performance only, optical path length in the range  $1.000 \pm 0.005$  cm. With distilled water in the cells, determine any absorbance differences.

#### D-2.11 Spectrophotometer

Spectral range 250 to 400 nm with spectral slit width of 2 nm or less; under instrument operating conditions for these absorbance measurements, the spectrophotometer shall also meet the following performance requirements:

Absorbance repeatability,  $\pm 0.01$  at 0.4 absorbance.

Absorbance accuracy\*,  $\pm 0.05$  at 0.4 absorbance.

Wavelength repeatability,  $\pm 0.2$  nm.

Wavelength accuracy,  $\pm 1.0$  nm.

\*As determined by procedure using potassium chromate for reference standard. The accuracy is to be determined by comparison with standard values at 290, 345 and 400 nm.

#### D-2.12 Nitrogen Cylinder

Water pumped or equivalent purity nitrogen in cylinder equipped with regulator and valve to control flow at  $0.351 \text{ 5 kg/cm}^2$ .

### D-3 REAGENTS AND MATERIALS

**D-3.1** Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

#### D-3.2 Organic Solvents

All solvents used throughout the procedure shall meet the specifications and tests described in this specification. The iso-octane, benzene, acetone, and methyl alcohol designated in the list following this paragraph shall pass the tests mentioned below:

- a) To the specified quantity of solvent in a 250 ml Erlenmeyer flask, add 1 ml of purified n-hexadecane and evaporate on the steam bath under a stream of nitrogen (a loose aluminium foil jacket around the flask will speed evaporation). Discontinue evaporation when not over 1 ml of residue remains (to the residue from benzene add a 10 ml portion of purified iso-octane, re-evaporate and repeat once to ensure complete removal of benzene).
- b) Alternatively, the evaporation time can be reduced by using the optional evaporation flask. In this case, the solvent and n-hexadecane are placed in the flask on the steam bath, the tube assembly is inserted, and a stream of nitrogen is fed through the inlet tube while the outlet tube is connected to a solvent trap and vacuum line in such a way as to prevent any flow-back of condensate into the flask.
- c) Dissolve the 1 ml of hexadecane residue in iso-octane and make up to 25 ml in volume. Determine the absorbance in the 5 cm path length cell compared to iso-octane as reference. The absorbance of the solution of the solvent residue (except for methyl alcohol) shall not exceed 0.01 per cm path length between 280 and 400 nm. For methyl alcohol this absorbance value shall be 0.00.

##### D-3.2.1 Iso-octane (2, 2, 4-Trimethylpentane)

Use 180 ml for the test described in the preceding paragraph. Purify, if necessary, by passing it through a column of activated silica gel above 90 cm in length and 5 to 8 cm in diameter.

##### D-3.2.2 Benzene

Use 150 ml for the test. Purify, if necessary, by distillation or otherwise.



**D-3.2.3 Acetone**

Use 200 ml for the test. Purify, if necessary, by distillation.

**D-3.2.4 Eluting Mixtures**

- a) *10 Percent Benzene in Iso-octane* — Pipet 50 ml of benzene into a 500 ml glass stoppered volumetric flask and adjust to volume with iso-octane, with mixing.
- b) *20 Percent Benzene in Iso-octane* — Pipet 50 ml of benzene into a 250 ml glass stoppered volumetric flask and adjust to volume with iso-octane, with mixing.
- c) *Acetone-benzene-water* — Add 20 ml of water to 380 ml of acetone and 200 ml of benzene, and mix.

**D-3.2.5 n-Hexadecane, 99 Percent, Olefin-free**

Dilute 0.1 ml of n-hexadecane to 25 ml with iso-octane and determine the absorbance in a 5 cm cell compared to iso-octane as reference point between 280 to 400 nm. The absorbance per centimeter path length shall not exceed 0.00 in this range. Purify, if necessary by percolation through activated silica gel or by distillation.

**D-3.2.6 Methyl Alcohol**

Use 10.0 ml of methyl alcohol. Purify, if necessary, by distillation.

**D-3.3 Dimethyl Sulphoxide**

Pure grade, clear, water white (m.p.18°C, *Min*). Dilute 120 ml of dimethyl sulphoxide with 240 ml of distilled water in a 500 ml separatory funnel, mix and allow cooling for 5 to 10 min. Add 40 ml of iso-octane to the solution, extract by shaking the funnel vigorously for 2 min. Draw off the lower aqueous layer into a second 500 ml separatory funnel, and repeat the extraction with 40 ml of iso-octane. Draw off and discard the aqueous layer. Wash each of the 40 ml extractives three times with 50 ml portions of distilled water. Shaking time for each wash is 1 min. Discard the aqueous layers. Filter the first extractive through anhydrous sodium sulphate prewashed with iso-octane (*see D-3.2* for preparation of filter) in to a 250 ml Erlenmeyer flask, or optionally into the evaporating flask. Wash the first separatory funnel with the second 40 ml iso-octane extractive, and pass through the sodium sulphate into the flask. Then wash the second and first separatory funnels successively with a 10 ml portion of iso-octane, and pass the solvent through the sodium sulphate into the flask. Add 1 ml of n-hexadecane and evaporate the iso-octane on the steam bath under nitrogen. Discontinue evaporation when not over 1ml of residue remains. To the residue, add a 10 ml portion of iso-octane and re-evaporate to 1 ml of hexadecane. Again, add 10 ml of iso-octane to the residue and evaporate to 1 ml of hexadecane to ensure

complete removal of all volatile materials. Dissolve the 1 ml of hexadecane in iso-octane and make up to 25 ml in volume. Determine the absorbance in 5 cm path length cells compared to iso-octane as reference. The absorbance of the solution should not exceed 0.02 per centimeter path length in the 280 - 400 nm range (*see Note*).

Purify, if necessary, by the following procedure:

- a) To 1 500 ml of dimethyl sulphoxide in a 2 litre glass stoppered flask, add 6.0 ml of phosphoric acid and 50 g of Norit A (decolorizing carbon, alkaline or equivalent). Stopper the flask, and with the use of a magnetic stirrer (tetrafluoroethylene polymer coated bar) stir the solvent for 15 min. Filter the dimethyl sulphoxide through four thickness of fluted paper. If the initial filtrate contains carbon fines, re-filter through the same filter until a clear filtrate is obtained.
- b) Protect the sulphoxide from air and moisture during this operation by covering the solvent in the funnel and collection flask with a layer to iso-octane. Transfer the filtrate to a 2 litre separatory funnel, draw off the dimethyl sulphoxide into the 2 litre distillation flask of the vacuum distillation assembly, and distill at approximately 3 mm Hg pressure or less. Discard the first 200 ml fraction of the distillate and replace the distillate collection flask with a clean one. Continue the distillation until approximately 1 litre of the sulphoxide has been collected.
- c) At completion of the distillation, the reagent should be stored in glass stoppered bottles since, it is very hygroscopic and will react with some metal containers in the presence of air.

NOTE — Difficulty in meeting this absorbance specification may be due to organic impurities in the distilled water. Repetition of the test omitting the dimethyl sulphoxide will disclose their presence. If necessary to meet the specification, purify the water by re distillation, passage through an ion-exchange resin, or otherwise.

**D-3.4 Phosphoric Acid** — 85 percent.

**D-3.5 Sodium Borohydride** — 98 percent.

**D-3.6 Magnesium Oxide**

Place 100 g of the magnesium oxide in a large beaker, add 700 ml of distilled water to make a thin slurry and heat on a steam bath for 30 min with intermittent stirring. Stir well initially to ensure that all the adsorbent is completely wetted. Using a buchner funnel and a filter paper of suitable diameter, filter with suction. Continue suction until water no longer drips from the funnel. Transfer the adsorbent to a glass trough lined with aluminium foil (free from rolling oil). Break up the magnesia with a clean spatula and spread out the adsorbent on the aluminium foil in a layer of about

1 to 2 cm thick. Dry for 24 h at  $160 \pm 1^\circ\text{C}$ . Pulverize the magnesia with mortar and pestle. Sieve the pulverized adsorbent between 60 to 180 mesh. Use the magnesia retained on the 180 mesh sieve.

#### D-3.7 Celite 545

#### D-3.8 Magnesium Oxide-Celite 545 Mixture (2+1) by Mass

Place the magnesium oxide (60 to 180 mesh) and the celite 545 in 2 to 1 proportions respectively, by mass in a glass stoppered flask large enough for adequate mixing. Shake vigorously for 10 min. Transfer mixture to a glass trough lined with aluminium foil (free from rolling oil) and spread it out on a layer about 1 to 2 cm thick. Reheat the mixture at  $160 \pm 1^\circ\text{C}$  for 2 h, and store in a tightly closed flask.

#### D-3.9 Sodium Sulphate, Anhydrous, Preferably in Granular Form

For each bottle of sodium sulphate reagent used, establish as follows the necessary sodium sulphate:

Prewash to provide such filters required in the method. Place approximately 35 g of anhydrous sodium sulphate in a 30 ml coarse, fritted glass funnel or in a 65 mm filter funnel with glass wool plug. Wash with successive 15 ml portions of the indicated solvent until a 15 ml portion of the wash shows 0.00 absorbance per cm path length between 280 and 400 nm when tested as prescribed under **D-3.2**. Usually three portions of wash solvent are sufficient.

**D-3.9.1** Before proceeding with analysis of a sample, determine the absorbance in a 5 cm path cell between 250 and 400 nm for the reagent blank by carrying out the procedure, without a wax sample, at room temperature, recording the spectra after the extraction stage and after the complete procedure as prescribed. The absorbance per cm path length following the extraction stage should not exceed 0.040 in the wavelength range from 280 to 400 nm. The absorbance per centimeter path length following the complete procedure should not exceed 0.070 in the wavelength range 280 to 299 nm inclusive, nor 0.045 in the wavelength range from 300 to 400 nm. If in either spectrum the characteristic benzene peaks in the 250 to 260 nm region are preset, remove the benzene by the procedure described under **D-3.2** and record absorbance again.

**D.3.9.2** Place 300 ml of dimethyl sulfoxide in a 1 litre separatory funnel and add 75 ml of phosphoric acid. Mix the contents of the funnel and allow to stand for 10 min (the reaction between the sulfoxide and the acid is exothermic. Release pressure after mixing, then keep funnel stoppered). Add 150 ml of iso-octane and shake to pre-equilibrate the solvents. Draw off the individual layers and store in glass stoppered flasks.

### D-4 PROCEDURE

**D-4.1** Place a representative 1 kg sample of wax, or if this amount is not available, the entire sample, in a beaker of a capacity about three times the volumes of the sample and heat with occasional stirring on a steam bath until the wax is completely melted and homogeneous. Weigh four  $25 \pm 0.2\text{g}$  portions of the melted wax in separate 100 ml beakers. Reserve three of the portions for later replicate analysis as necessary. Pour one weighed portion immediately after re-melting (on the steam bath) into a 500 ml separatory funnel containing 100 ml of the pre-equilibrated sulfoxide-phosphoric acid mixture that has been heated in the heating jacket at a temperature just high enough to keep the wax melted (*see Note*).

NOTE — In preheating the sulfoxide acid mixture, remove the stopper of the separatory funnel at intervals to release the pressure.

**D-4.2** Promptly complete the transfer of the sample to the funnel in the jacket with portions of the pre-equilibrated iso-octane warming the beaker, if necessary, and using a total volume of just 50 ml of the solvent. If the wax comes out of solution during these operations, let the stoppered funnel remain in the jacket until the wax re-dissolves. (Remove stopper from the funnel at intervals to release pressure). When the wax is in solution, remove the funnel from the jacket and shake it vigorously for 2 min. Set up three 250 ml separatory funnels with each containing 30 ml of pre-equilibrated iso-octane. After separation of the liquid phases, allow to cool until the main portion begins to show a precipitate. Gently swirl the funnel when precipitation first occurs on the inside surface of the funnel to accelerate this process. Carefully draw off the lower layer, filter it slowly through a thin layer of glass wool fitted loosely in a filter funnel into the first 250 ml separatory funnel, and wash in tandem with the 30 ml portions of iso-octane contained in the 250 ml separatory funnels. Shaking time for each wash is 1 min. Repeat the extraction operation with two additional portions of the sulfoxide acid mixture, replacing the funnel in the jacket after each extraction to keep the wax in solution and washing each extractive in tandem through the same three portions of iso-octane.

**D-4.3** Collect the successive extractives (300 ml total) in a separatory funnel (preferably 2litre), containing 480 ml or distilled water, mix and allow to cool for a few min after the last extractive has been added. Add 80 ml of iso-octane to the solution, extract by shaking the funnel vigorously for 2 min. Draw off the lower aqueous layer into a second separatory funnel (preferably 2 litre), and repeat the extraction with 80 ml of iso-octane. Draw off the lower aqueous layer. Wash each of the 80 ml extractives three times with 100 ml portions of distilled water. Shaking time for



each wash is 1 min. Discard the aqueous layers. Filter the first extractive through anhydrous sodium sulphate prewashed with iso-octane (*see D-3.2* for preparation of filter) into a 250 ml Erlenmeyer flask (or optionally into the evaporation flask). Wash the first separatory funnel with the second 80 ml iso-octane extractive and pass through the sodium sulphate. Then wash the second and first, separatory funnels successively with a 20 ml portion of iso-octane and pass the solvent through the sodium sulphate into the flask. Add 1 ml of n-hexadecane and evaporate the iso-octane on the steam bath under nitrogen. Discontinue evaporation when not over 1 ml of residue remains. To the residue, add a 10 ml portion of iso-octane, re-evaporate to 1 ml of hexadecane, and repeat this operation once.

**D-4.4** Quantitatively transfer the residue with iso-octane to a 25 ml volumetric flask, make up to the volume, and mix. Determine the absorbance of the solution in the 5 cm path length cells compared to iso-octane as reference between 280 to 400 nm (take care to lose none of the solution in filling the sample cell). Correct the absorbance values for any absorbance of the solution in the 5 cm path length cell compared to iso-octane as reference between 280 to 400 nm (take care to lose none of the solution in filling the sample cell). Correct the absorbance values for any absorbance derived from reagents as determined by carrying out the procedure without a wax sample. If the corrected absorbance does not exceed the limit prescribed in Table 1, the wax meets the ultraviolet absorbance specifications. If the corrected absorbance per cm path length exceeds the limits prescribed in Table 1, proceed in accordance with **D-4.5** and **D-4.6**.

**D-4.5** Quantitatively transfer the iso-octane solution to a 125 ml flask equipped with 24/40 joint and evaporate the iso-octane on the steam bath under a stream of nitrogen to a volume of 1 ml of hexadecane. Add 10 ml of methyl alcohol and approximately 0.3 g of sodium borohydride. (Minimize exposure of the borohydride to the atmosphere. A measuring dipper may be used). Immediately fit a water-cooled condenser equipped with a 24/40 joint and with a drying tube into the flask, mix until the borohydride is dissolved, and allow to stand for 30 min at room temperature, with intermitted swirling. At the end of this period, disconnect the flask and evaporate the methyl alcohol on the steam bath under nitrogen until the sodium borohydride begins to come out of the solution. Then add 10 ml of iso-octane and evaporate to a volume of about 2 to 3 ml. Again, add 10 ml of iso-octane and concentrate to a volume of approximately 5 ml. Swirl the flask repeatedly to assure adequate washing of the sodium borohydride residues.

**D-4.6** Fit the tetrafluoroethylene polymer disc on the upper part of the stem of the chromatographic tube, then place the tube with the disc on the suction flask

and apply the vacuum (approximately 135 mm Hg pressure). Weigh out 14 g of the 2 : 1 magnesium oxide - celite 545 mixture and pour the adsorbent mixture into the chromatographic tube in approximately 3 cm layers. After the addition of each layer, level off the top of the adsorbent with a flat glass rod or metal plunger by pressing down firmly until the adsorbent is well packed. Loosen the top most few mm of each adsorbent layer with the end of a metal rod before the addition of the next layer. Continue packing in this manner until all the 14 g of the adsorbent is added to the tube. Level off the top of the adsorbent by pressing down firmly with a flat glass rod or metal plunger to make the depth of the adsorbent bed approximately 12.5 cm in depth. Turn off the vacuum and remove the suction flask. Fit the 500 ml reservoir onto the top of the chromatographic column and pre-wet the column by passing 100 ml of iso-octane. Adjust the nitrogen pressure so that the rate of descent of the iso-octane coming off the column is between 2 to 3 ml per min. Discontinue pressure just before the last of the iso-octane reaches the level of the adsorbent.

**CAUTION** — Do not allow the liquid level to recede below the adsorbent level at any time.

Remove the reservoir, decant the 5 ml iso octane concentrate solution into the column, and with slight pressure again allow the liquid level to recede to barely above the adsorbent level to recede to barely above the adsorbent level. Rapidly complete the transfer similarly with two 5 ml portions of iso-octane, swirling the flask repeatedly each time to assure adequate washing of the residue. Just before the final 5 ml, wash reaches the top of the adsorbent, add 100 ml of iso-octane to the reservoir and continue the percolation at 2 to 3 ml per min rate. Just before the last of the iso-octane reaches the adsorbent level, add 100 ml of 10 percent benzene in iso-octane to the reservoir and continue the percolation at the above mentioned rate. Just before the solvent mixture reaches adsorbent level, add 25 ml of 20 percent benzene in iso-octane to the reservoir and continue the percolation at 2 to 3 ml per min until all this solvent mixture was removed from the column. Discard all the elution solvents collected up to this point. Add 300 ml of the acetone-benzene water mixture to the reservoir and percolate through the column to elute the polynuclear compounds. Collect the elute in a clean 1 litre separatory funnel. Allow the column to drain until most of the solvent mixture is removed. Wash the elute three times with 300 ml portions of distilled water, shaking well for each wash. (The addition of small amounts of sodium chloride facilitates separation). Discard the aqueous layer after each wash. After the final separation, filter the residual benzene through anhydrous sodium sulphate prewashed with benzene (*see D-3.9* for preparation of filter) into a 250 ml Erlenmeyer flask (or optionally into

the evaporation flask). Wash the separatory funnel with two additional 20 ml portions of benzene, which are also filtered through the sodium sulphate. Add 1 ml of n-hexadecane and completely remove the benzene by evaporation under nitrogen, using the special procedure to eliminate benzene as previously described under **D-3.2**. Quantitatively transfer the residue with iso-octane to a 25 ml volumetric flask and adjust to volume. Determine the absorbance of the solution in the 5cm path length cells compared to iso-octane as reference between 200 to 400 nm. Correct for any absorbance

derived from the reagents as determined by carrying out the procedure without a wax sample. If either spectrum shows the characteristic benzene peaks in the 250 to 260 nm region, evaporate the solution to remove benzene by the procedure under **D-3.3**. Dissolve the residue, transfer quantitatively, and adjust to volume in iso-octane in a 25 ml volumetric flask. Record the absorbance again. If the corrected absorbance does not exceed the limits prescribed in Table 1, the wax meets the ultraviolet absorbance specifications.

## ANNEX E

[ Table 1, Sl No. (ix) ]

### DETERMINATION OF ODOUR

#### E-1 GENERAL

**E-1.1** In some uses of petroleum wax, such as food packaging, odour intensity of the wax is an important property. For example, some description of limits on wax odour often appears in specification for petroleum wax. The method given here provides a basis for agreement between laboratories on the odour intensity of wax using a numerical scale rather than descriptive terms. While the method is primarily intended for rating odour intensity, results may be influenced by odour type.

**E-1.2** This method describes a procedure for rating the odour intensity of paraffin wax.

#### E-2 TERMINOLOGY

For the purpose of this test, the following definition shall apply.

##### E-2.1 Odour of a Paraffin Wax

Numerical rating corresponding to the odour scale description that best fits the sample being tested.

#### E-3 OUTLINE OF THE METHOD

Odour test specimens are prepared from paraffin wax by placing approximately 10 g of thin shavings on odour-free paper or glassine. Individual test specimens are then evaluated for odour by each panel member and assigned the number corresponding to the odour scale description best fitting the intensity of the odour. As an alternative procedure, the wax shavings are placed in bottles, with each panel member making the odour evaluating between 15 and 60 min after the specimens are prepared. The average of the panel rating is reported as the odour rating of the sample.

#### E-4 APPARATUS

##### E-4.1 Scraper

A knife, vegetable scraper or other sharp instrument to produce thin shaving from wax and which may be easily cleaned.

##### E-4.2 Paper

Odour free or glassine for receiving the wax shavings.

##### E-4.3 Bottles

Wide mouth, with caps.

#### E-5 TEST PANEL

Test panel shall consist of at least five persons as agreed to between the purchaser and the supplier. Individual panel members shall not evaluate a series of more than three specimens at a time. Intervals of at least 15 min between each series shall be mandatory.

#### E-6 SAMPLE AND TEST SPECIMEN

**E-6.1** The sample shall consist of a block of wax at room temperature, from which at least 100 g of shavings may be obtained.

**E-6.2** The test specimen for evaluation by each panel member shall consist of approximately 10 g wax in thin shavings obtained from the sample.

#### E-7 PROCEDURE

**E-7.1** The test shall be conducted in a room, free from odour and having normal relative humidity. Any of the two methods described in **E-7.2** and **E-7.3** shall be followed to observe the odour of the wax. Panel

members shall not discuss the results until all tests have been completed on a sample.

### E-7.2 Method A

**E-7.2.1** Scrape the surface of the sample to remove any foreign material and discard those scrapings. Using a clean scraper, prepare test specimens of approximately 10 g each of wax in thin shavings, placing them on odour-free paper or glassine. Obtain shavings, which represent a cross section of the sample being tested. Prepare one test specimen for each panel member.

**E-7.2.2** Each panel member shall make the odour evaluation immediately after the shavings are prepared by placing the nostrils in contact with the shavings and sniffing lightly (*see* Note under E-7.3.1).

### E-7.3 Method B

**E-7.3.1** Immediately after preparing, the shavings transfer them to clean, odour-free bottles that are then capped. Prepare one bottle for each panel member. Each panel member shall then evaluate the test specimen between 15 and 60 min after preparation, by removing the bottle cap, placing the nostrils at the bottle mouth and sniffing lightly (*see* Note).

NOTE — If the specimen is smelled repeatedly, the odour usually seems to decrease due to the olfactory fatigue or less of volatile components in the specimen or both.

**E-7.3.2** Record the odour of specimen in terms of numerical rating shown below, which corresponds to the odour intensity that best characterizes each specimen:

<i>Numerical rating</i>	<i>Odour description</i>
0	None
1	Slight
2	Moderate
3	Strong
4	Very strong

## E-8 CALCULATION AND REPORTING

**E-8.1** Calculate to the nearest unit average of numerical ratings given to sample by the panel members. If any of the individual ratings differ from the average by more than 1 unit, repeat the test for the sample by all panel members until the individual ratings agree with the average rating within  $\pm 1$  unit.

**E-8.2** Report the average odour rating calculated to the nearest unit as the odour rating of the sample.

## ANNEX F

( Clause B-3.1)

### METHOD OF TEST FOR MEASUREMENT OF MAXIMUM PORE DIAMETER OF RIGID POROUS FILTERS

#### F-1 GENERAL

This method describes a test procedure for the determination of the acceptability of porous filter sticks. This method establishes the maximum pore diameter and provides a means of detecting and measuring changes, which occur from continued use.

#### F-2 TERMINOLOGY

For the purpose of this test, the following definition shall apply.

##### F-2.1 Maximum Pore Diameter

It is the diameter in microns of the largest opening in filter.

NOTE — It is recognized that the maximum pore diameter as defined herein does not necessarily indicate the physical dimensions of the largest pore in the filter. It is further recognized that the pores are highly irregular in shape. Because

of the irregularity in shape and other phenomena characteristic of filtration, a filter may be expected to retain all particles larger than the maximum pore diameter as defined and determined herein, and will generally retain particles, which are much smaller than the determined diameter.

#### F-3 OUTLINE OF METHOD

The filter is cleaned and wetted with water. It is then immersed in water and air pressure is applied against its upper surface until the first bubble or air passes through the filter. The maximum pore diameter is calculated from the surface tension of water and the applied pressure.

#### F-4 APPARATUS

##### F-4.1 Manometer

Mercury filled and readable to 0.5 mm.

#### F-4.2 Air Supply

Clean and filtered.

#### F-4.3 Pressure Regulator

Needle-valve type.

#### F-4.4 Drying Oven

### F-5 PROCEDURE

**F-5.1** Clean the filter sticks by soaking in concentrated hydrochloric acid and then wash them with distilled water. Rinse with acetone, air dry, and place in drying oven at 105°C for 30 min.

**F-5.2** Thoroughly wet the clean filter to be tested by soaking it in distilled water.

**F-5.3** Assemble the apparatus as shown in Fig. 6. Apply pressure slowly from a source of cleaned air.

**F-5.4** Immerse the filter just below the surface of the water.

NOTE — If a head of liquid exists above the surface of the filter, back pressure produced shall be deducted from the observed pressure.

**F-5.5** Increase the air pressure to 10 mm below the acceptable pressure limit and then at a slow uniform rate of about 3 mm of mercury per min until the first bubble passes through the filter. This can be conveniently observed by placing the beaker or test tube over a mirror. Read the manometer when the first bubble passes off the underside of the filter.

### F-6 CALCULATION

The pore diameter is calculated as follows:

$$D = \frac{2180}{P}$$

Where,

$D$  = pore diameter, in micron; and

$P$  = manometer reading, in millimeter of mercury.

NOTE — From this equation, pressure corresponding to the upper and lower limits of the specified pore diameters can be calculated. These pressures may be used for acceptance testing.

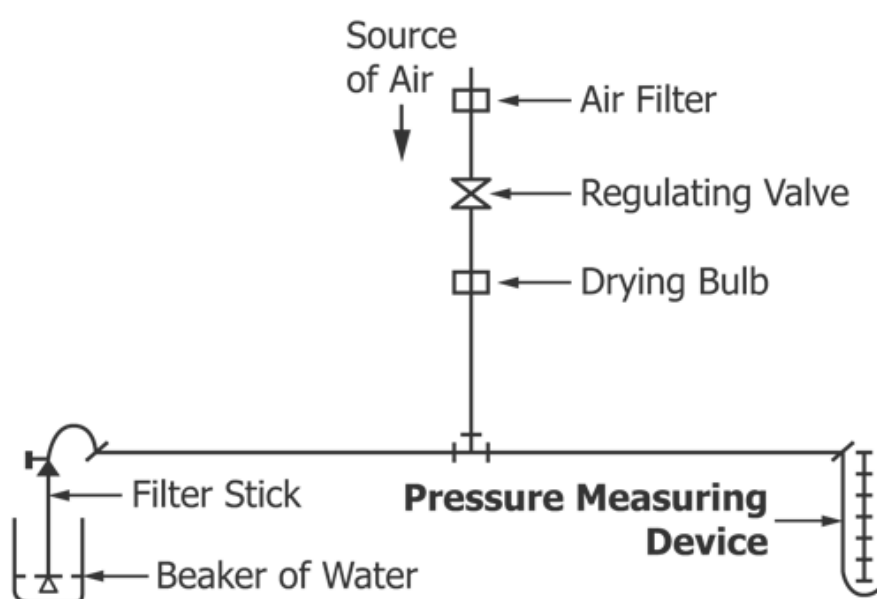


FIG. 6 ASSEMBLY OF APPARATUS FOR CHECKING PORE DIAMETER OF FILTER STICK

**ANNEX G***( Foreword )***COMMITTEE COMPOSITION**

Petroleum and Their Related Products of Synthesis or Biological Origin Sectional Committee, PCD 03

<i>Organization</i>	<i>Representative(s)</i>
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